

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application of
Maria Anna Wubben et al.
Serial No. 08/776,321 Examiner: C.E. Sherrer
Filed: April 15, 1997 Art Unit: 1761
For: PECTINS AS FOAM STABILIZERS FOR BEVERAGES HAVING A
FOAM HEAD

DECLARATION of Alexandra J.M. Wijsman

Alexandra Johanna Mathilda Wijsman herewith declares as follows:

1. I presently reside at Hondsdraveld 23, 3448 EC Woerden, the Netherlands.

2. I am an employee of Heineken Technical Services B.V. at Zoeterwoude, the Netherlands, the assignee of the present invention. I am a graduate of the Agricultural University of Wageningen, with a specialization in food technology. Since 1995 I have been involved in research on raw materials for the production of beer, such as polysaccharides.

3. In order to determine the effect of the amount of pectin on foam stability of beer, an experiment was conducted using different amounts of hop-pectin. I conducted part of the experiment; the other parts were conducted in my presence. The experiments and results are described as follows.

Pectin preparations (extract from CO₂ extract residues) were obtained by an extraction procedure using the same principles as described in the patent application on page 14, however, modified to take into account industrial size unit operations.

The extract contained 42.9 % w/w of pectin (= anhydrogalacturonic acid), the remainder being mainly sugars and minerals, and a small amount of denatured (insoluble) protein (8% w/w) and phenolic compounds (2% w/w).

4. Hop pectin preparations were dissolved in water before being added to reference beer (pilsner beer) in amounts of 1, 5, 10, 15, 20, 25 and 30 g pectin per hectolitre of beer (based on actual pectin (anhydrogalacturonic acid, AUA) content). For the amounts of 1, 5 and 10 g/hl the pectin preparations were dissolved in 5 ml of water and the other amounts were dissolved in 10 ml of water. The bottles were shaken for 48 hours at room temperature. The foam stability was then determined using the Nibem foam meter.

5. The results of the test were as follows.

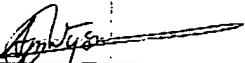
Foam stability of beer after addition of several quantities of hop pectin
AUA content hop pectin: 42.9% w/w

| Samples | Pectin addition g pectin preparation/ hl | Pectin addition as AUA g AUA / hl | Foam stability (sec) | | Foam improvement (sec) |
|------------|---|--------------------------------------|----------------------|---------|---------------------------|
| | | | average | average | |
| Reference | - | - | 289 | 285 | 287 |
| Hop pectin | 2.3 | 1 | 327 | 324 | 326 |
| Hop pectin | 11.7 | 5 | 357 | 354 | 356 |
| Hop pectin | 23.3 | 10 | 380 | 382 | 381 |
| Hop pectin | 35.0 | 15 | 456 | 422 | 439 |
| Hop pectin | 46.6 | 20 | 480 | 410 | 445 |
| Hop pectin | 58.3 | 25 | 499 | 479 | 489 |
| Hop pectin | 69.9 | 30 | 473 | 477 | 475 |

6. The undersigned declares that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that wilful false statements so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such wilful false statements so made may jeopardize the validity of the document, or application, or any patent issuing thereon.

Signed this 29 day of October, 1999

By


Alexandra J.M. Wijsman

22.NOV.1999 15:35

HEINEKEN HTS R&D

NO.414

P.2/5

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application of
Maria Anna Wubben et al.
Serial No. 08/776,321
Filed: April 15, 1997
For: PECTINS AS FOAM STABILIZERS FOR BEVERAGES HAVING A
FOAM HEAD

Examiner: C.E. Sherrer
Art Unit: 1761

DECLARATION of Albert Doderer

Albert Doderer herewith declares as follows:

1. I presently reside at Albast 47, 2719 TV
Zoetermeer, the Netherlands.

2. I am an employee of Heineken Technical
Services B.V. at Zoeterwoude, the Netherlands, the assignee
of the present invention. I am a graduate of the Free
University Amsterdam with a specialisation in
biology/biochemistry. Since 1985 I have been involved in
research on raw materials for the production of beer. I am a
co-inventor of the present invention.

3. In the period the present invention was made,
prior to August 1994, various experiments were conducted with
hop pectins extracted from several hop varieties and several
parts of hop plants. In addition, the influence of the amount
of hop pectin on beer was investigated.

Pectin preparations (extracts) were obtained by the
extraction procedure described in the patent application on
page 14. The hop pectin preparations, which were added, were
not 100% pectin. Rather, the preparations were approximately
55-75% pectin (as anhydrogalacturonic acid); see page 17,
Annex 6, of the patent application.

22.NOV.1999 15:35

HEINEKEN HTS R&D

NO.414

P.3/5

Hop pectin preparations were dissolved in 5 ml water before being added to reference beer (pilsner beer) in amounts which resulted in the addition of approximately 3 to 7 grams of pectin per hectolitre of beer. The bottles were shaken for 60 hours at room temperature. The foam stability was then determined using the Nibem foam meter.

The experiments were done either by me, or under my direct supervision.

The results of these experiments were included in the above-referenced patent application, and a summary of the most relevant ones is given in the following table (see the table below).

22.NOV.1999 15:36

HEINEKEN HTS R&D

NO.414

P.4/5

Results of foam stability of pilsner reference beer after addition of hop pectin derived from bines, cones or waste of four different hop varieties.

| Samples | g pectin preparation/l | Pectin addition as AUA g AUA/l | Foam stability (sec) | Foam improvement (sec) |
|------------------------|------------------------|-----------------------------------|-------------------------|---------------------------|
| Hersbrucker | | | | |
| bine | 5 | 3.3 | 348 | 42 |
| | 10 | 6.6 | 354 | 50 |
| cone | 5 | 9.05 | 351 | 47 |
| | 10 | 6.1 | 365 | 61 |
| waste | 5 | 2.75 | - | - |
| | 10 | 5.5 | 345 | 41 |
| Aroma perlé | | | | |
| bine | 5 | 3.75 | 343 | 39 |
| | 10 | 7.5 | 374 | 70 |
| cone | 5 | 3.25 | 342 | 38 |
| | 10 | 8.5 | 366 | 62 |
| waste | 5 | 2.8 | 339 | 35 |
| | 10 | 5.6 | 351 | 47 |
| Northern brewer | | | | |
| cone | 5 | 2.8 | 345 | 42 |
| | 10 | 5.6 | 360 | 56 |
| waste | 5 | 3.6 | 334 | 30 |
| | 10 | 7.2 | 380 | 56 |
| Brewers gold | | | | |
| bine | 5 | 3.2 | 336 | 32 |
| | 10 | 6.4 | 368 | 64 |
| cone | 5 | 3.0 | 341 | 37 |
| | 10 | 6.0 | 359 | 55 |
| waste | 5 | 3.25 | 343 | 39 |
| | 10 | 8.5 | 352 | 48 |
| Reference | - | - | 304 | - |

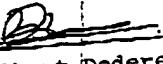
4. At that time the results of these experiments were surprising and unexpected. It was surprising and unexpected that the addition of hop pectin would improve the foam stability of reference beer in the dramatic fashion shown by the test results above.

22.NOV.1999 15:36

HEINEKEN HTS R&D

5. The undersigned declares that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that wilful false statements so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such wilful false statements so made may jeopardize the validity of the document, or application, or any patent issuing thereon.

Signed this 22nd day of November, 1999

By 
Albert Doderer

OFFICIAL